



Short communication

The electrochemical behavior of three air cathodes for microbial electrochemical system (MES) under meter scale water pressure

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H I G H L I G H T S

- A 3 m test system was built to investigate the effect of water pressure.
- The cathodes of Pt-CC, Pt-CM, and AC-MM showed various responses under pressure.
- The AC-MM functioned stable and achieved the best water pressure tolerance.
- The mechanical properties of cathodes close related to their performance responses.

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To produce cathodes with high water pressure tolerance for the practical application of microbial electrochemical system (MES), a 3-m test configuration is set up. Three kinds of cathodes, including Pt-CC (carbon cloth with platinum carbon), Pt-CM (carbon mesh with platinum carbon), and AC-MM (metal mesh with activated carbon), are investigated. The electrochemical performances of these cathodes are tested with linear sweep voltammetry under varied water pressures. Current densities of Pt-CC and Pt-CM increase with the rising water pressures till the maximum endurable water head, which are 100 cm for Pt-CC and 130 cm for Pt-CM. Yet electrochemical performances of AC-MM remained stable under the tested water pressure range from 0 to 30 KPa. The deformation of cathodes under varied water pressures causes the changes of cathode performances. The curvature degrees of cathodes relate to their mechanical properties. Elastic modulus of AC-MM is $4 \pm 0.4 \times 10^3$ MPa, which is over 10 times larger than that of Pt-CM and over 60 times larger than that of Pt-CC. The best mechanical properties prevent AC-MM from the substantial deformation and the consequent lacerations and water flooding of diffusion layers.

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1. Introduction

Microbial electrochemical system (MES) is considered as a promising technology for wastewater treatment [1]. In MES, organic pollutants were oxidized by anode exoelectrogens, and the released electrons were finally combined with electron acceptors at the cathode [2,3]. Great efforts have been made in the past decade to increase the maximum power density by optimizing the reactor structure [4], constructing the energy harvest external circuit [5], and enhancing the performance of electrodes [6,7]. However, there

remained some challenges when considering the scaling up and the practical application of MES. Till now, most of the scaled-up systems were associated to the enlargement in liter-scale [4,8,9] and the height of MFC was in centimeter-level to decimeter-level [10]. The performances of cathodes were found to be affected under higher water pressure [11,12]. It is obviously important to enlarge the system to meter-scale in real application and the cathode could function stable under meter-level water pressure is of great significance.

Carbon cloth cathode with platinum catalyst (Pt-CC) was widely used for oxygen reduction [13] and carbon mesh cathode with Pt catalyst (Pt-CM) was used as a less expensive alternative of Pt-CC [6]. Recently, activated carbon cathode with metal mesh (AC-MM) was found to possess comparable catalytic properties with

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platinum carbon cathodes [14]. In these cathodes, hydrophilic catalysts layers were usually fixed on to the conductive supporting materials by various binders and diffusion layers were applied as thin layers using hydrophobic polymers, such as polytetrafluoroethylene (PTFE) [15] or polydimethylsiloxane (PDMS) [16]. These cathodes were widely investigated and optimized in previous works for the purpose of obtaining high power density output [14] and low manufacturing cost [6]. These air cathodes were considered to be promising in the practical application of MES, especially the carbon mesh (CM) cathode [10] and activated carbon cathode with stainless steel mesh (AC-MM) [17]. However, the impacts of water pressure on the performances of these cathodes were still unknown. In order to find out suitable cathodes for practical application, their maximum endurable water pressures should be examined.

In this study, to investigate the service life of different cathodes under different water pressures and to find out a suitable cathode with high water pressure tolerance, the cathodes of Pt-CC, Pt-CM and AC-MM were included as research targets. The relationships between cathode performances and the diffusion layer deformation under various water pressures, and mechanical properties of cathode structure were analyzed.

2. Experiment and methods

2.1. Test system

A 3-m test system was composed by two parts. The lower part was a 1.5 m height plexiglass rectangle column with an inner size of 4 cm (length) \times 4 cm (width). Fifteen pairs of cathode fixed sites were placed on both sides of the column. A plexiglass tube (Φ 3 cm)

was glued on the top of the plexiglass rectangle column to extend the test range from 1.5 m to 3 m (Fig. 1 and Fig. S1). The water pressures were calculated based on the height between the upper edge of the cathodes and the highest liquid surface. The effective diameter of the cathodes was 2 cm.

The 30% wet proofed carbon cloth (CC, type B, E-TEK), 10% PTFE wet proofed 3K carbon mesh (CM, Toray, 3K plain-weave carbon mesh) and corrosion resistant type 304 stainless steel mesh (SS, 60 mesh) were chosen as the conductive substrate of the tested cathodes followed previous reported methods [6,13,17]. The Pt-CC and Pt-CM were made followed the same process and coated with Pt/C catalyst layer with a loading rate of $0.5 \text{ mg Pt cm}^{-2}$. The activated carbon loading rate of AC-MM was 40 mg cm^{-2} . The PTFE loading rate in the diffusion layer of these cathodes was set at $25 \mu\text{L cm}^{-2}$ and the manufacturing processes were shown in Fig. 1.

2.2. Electrochemical analysis

The linear sweep voltammetry (LSV) was performed at a scan rate of 0.1 mV s^{-1} on the cathodes with a smooth bright Pt plate of 1 cm^2 apparent area as counter electrode and an Ag/AgCl reference electrode (195 mV vs. SHE, Spisic-Rex Instrument Factory, China) using a potentiostat (WonATech WMPG 1000, Korea). The test system was filled with 50 mM PBS (Na_2HPO_4 , 4.58 g L^{-1} ; $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$, 2.45 g L^{-1} ; conductivity of 6.9 mS cm^{-1}). The performances of cathodes under different water pressures were obtained by controlling water head in the test system. Prior to LSV test, the examined cathodes were stabilized under the certain water level for 24 h. The LSV tests under each water level were repeated until the curves obtained from three adjacent tests were overlapped. The LSV curves obtained in the last time were exhibited in this paper.

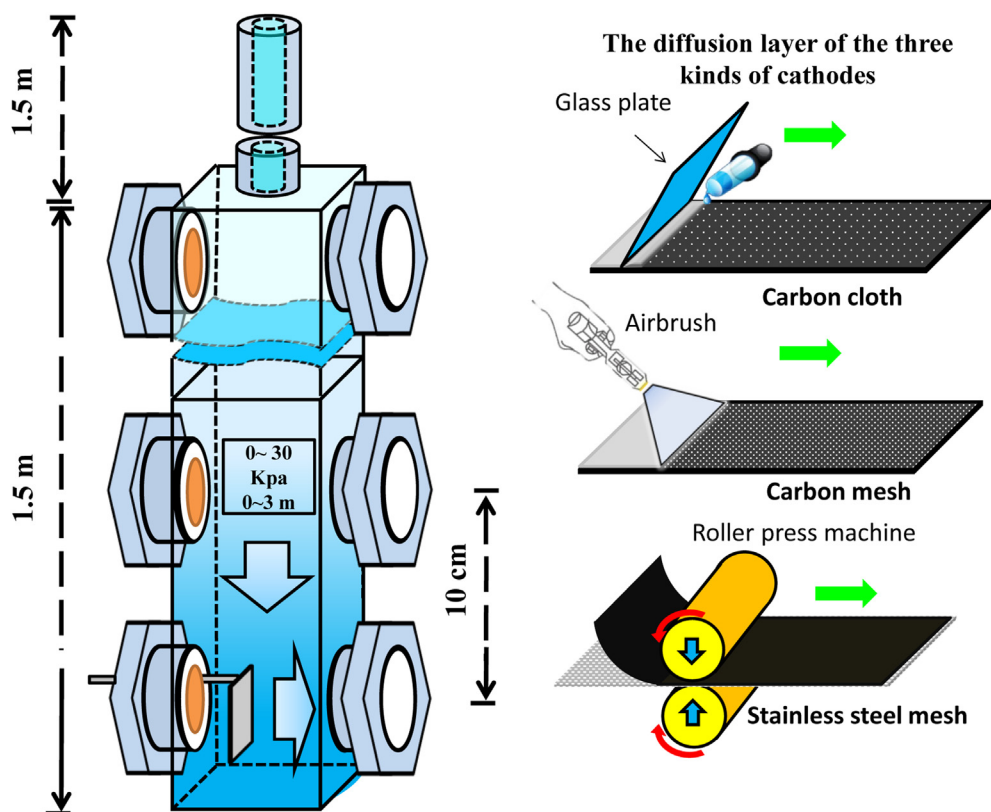


Fig. 1. Test system for water pressure influences on MES cathode performances (left) and schematic diagram of Diffusion layer fabrication for the three researched cathodes (right), smeared for Pt-CC, sprayed for Pt-CM and pressed for AC-MM.

The current densities of cathodes under 0 V (vs. SHE) were used to represent the overall trend of the LSV curves within the operation potential range of the investigated cathodes. The error bars (\pm SD) were calculated based on the average data of the three overlapped LSV curves.

2.3. The loopback tests

Loopback tests were designed to investigate whether the effects of water pressure on cathode performances were reversible. Due to the different water pressure resistant capability, cathodes were tested under different water pressure ranges (0–130 cm for Pt-CC, 0–180 cm for Pt-CM and 0–300 cm for AC-MM). The cathodes operated under their highest tested water head were retested under sequentially declining water levels, following the same procedure as described in 2.2.

2.4. Scanning electron micrograph imaging

The samples of Pt-CC, Pt-CM and AC-MM which had underwent water pressure of 130 cm, 180 cm and 300 cm were rinsed by deionized (DI) water and dried overnight at 40 °C in atmosphere oven. Prior to imaging, an electronically conductive Au coating was applied to the air sides (DL side) of each sample to reduce charging in the SEM. SEM were taken on a FEI Quanta 200 F (SEM, Hitachi Ltd. S-4700) [18]. To eliminate the influence of edge, the center part of the cathodes were used to represent the surface morphology changes.

2.5. Mechanical strength test

Mechanical strength characters, including modulus of elasticity, tensile stress and yield stress of cathodes, were tested on an instron 5569 electronic material testing system. The cathode samples tested for mechanical strength were cut to 10 × 1 cm. The samples were stretched to rupture on the testing system, during which the mechanical strength characters of all types of cathodes were obtained.

3. Results and discuss

3.1. Cathode performances under different water pressures

The linear sweep voltammetry (LSV) were performed to evaluate the electrochemical performances of the cathodes and the stability of their performances under various water pressures. The overall trends of the LSV curves were represented by the current densities of cathodes under 0 V (vs. SHE). This potential was chosen according to the highest power density point from the polarization curves of AC-MM cathodes in scaled-up reactors.

The LSV curves of Pt-CC and Pt-CM showed similar trends (Fig. 2 a-1 and b-1). Increasing water head from 0 to 40 cm, the current densities of the Pt-CC cathodes increased from $4.74 \pm 0.07 \text{ A m}^{-2}$ to $6.43 \pm 0.08 \text{ A m}^{-2}$, represented by the current densities of cathodes under 0 V (vs. SHE). The further increasing of water head from 40 cm to 100 cm made little contribution on the current (from $6.43 \pm 0.08 \text{ A m}^{-2}$ to $6.48 \pm 0.09 \text{ A m}^{-2}$). At the point of 100 cm, water droplets appeared on the Pt-CC surface and current density of Pt-CC dropped to $4.09 \pm 0.06 \text{ A m}^{-2}$ during the increasing of water head to 130 cm (Fig. 2 a-1). So the turning point of water pressure on Pt-CC was set as 100 cm.

In the loopback tests, the Pt-CC cathodes tested under the highest water head (130 cm) were reexamined under lower water head of 90 cm, 50 cm, 30 cm and 0 cm (Shown as R-90, R-50, R-30 and R-0 in Fig. 2 a-2), to investigate whether the influences of water

pressure on current density can be reversible. During the water head reduced from 130 cm to 0 cm, the current densities kept on dropping from $4.09 \pm 0.06 \text{ A m}^{-2}$ to $2.40 \pm 0.06 \text{ A m}^{-2}$ and water droplets outside of the diffusion layers existed until decreased to 30 cm. The carbon cloth cathodes were then washed by DI water and dried in stove under 40 °C and replaced on the test system. Under the circumstances, the current densities only recovered from $2.40 \pm 0.06 \text{ A m}^{-2}$ to $3.15 \pm 0.07 \text{ A m}^{-2}$ (showed as H-0 in Fig. 2, a-2), which indicated the reversible effects of water pressure on cathode performance.

For Pt-CM, current densities almost remained stable when water head increased from 0 cm to 60 cm. Yet, the current densities increased from 4.71 ± 0.08 to $6.49 \pm 0.11 \text{ A m}^{-2}$, while the water head rose from 60 cm to 130 cm. After that the current densities were slightly decreased during further increasing of water head to 180 cm from the turning point of 130 cm under potential lower than 0 V (vs. SHE) (Fig. 2 b-1). Water droplets were not observed under the tested water pressures.

The LSV curves in loopback tests were obtained for Pt-CM under various water head dropping from 180 to 0 cm (shown as R-140, R-100, R-60, R-30 and R-0 in Fig. 2 a-2). All the current densities under various water pressures in the loopback tests located in the area of $6.57 \pm 0.11 \text{ A m}^{-2}$ to $6.91 \pm 0.11 \text{ A m}^{-2}$. These current densities obtained were similar to that of the background test under the water pressure range of 130–180 cm (6.28 ± 0.11 – $6.87 \pm 0.10 \text{ A m}^{-2}$), but larger than the data under 0–130 cm (4.21 ± 0.08 – $6.49 \pm 0.11 \text{ A m}^{-2}$). Take 0 cm for instance, the current densities of Pt-CM in the loopback test was $6.63 \pm 0.09 \text{ A m}^{-2}$. Compared with the background data under 0 cm, the current densities were increased by about $2.0 \pm 0.3 \text{ A m}^{-2}$. The increased current densities in the loopback tests indicated that the improved cathode performances of Pt-CM under high water pressure can be maintained as an irreversible effect.

The current densities of AC-MM were kept at $7.2 \pm 0.3 \text{ A m}^{-2}$ (Fig. 2 c) under the whole test range (0–300 cm) and LSV curves showed good reproducibility. Within the test range, the exact turning point or the exact maximum endurable water pressure level of AC-MM was not found. Compared with Pt-CC and Pt-CM, water pressure has no significant impacts on AC-MM.

Obviously, the three cathodes exhibited different electrochemical responses under certain water pressures. The performances of Pt-CC and Pt-CM could be affected by water pressures. Minor performance increases of Pt-CC were observed under low level water pressures (lower than 40 cm). After that, the cathode performances kept stable (40–100 cm). However, under the water pressures higher than the turning points, irreversible negative effects appeared with the leaking of diffusion layer. The Pt-CM exhibited different responses to Pt-CC. The performances of the Pt-CM kept stable under low level water pressure (lower than 60 cm). After that, increased performances of Pt-CM were observed (60–130 cm). However, the positive effects of water pressure stopped and negative effects appeared after the turning point of 130 cm. The AC-MM functioned stable under the tested water range of 0–300 cm and achieved the best water pressure tolerance.

3.2. The micrograph of diffusion layer on three cathodes

To investigate the inherent reason of different water endurable abilities of the cathodes, the micrograph images of the diffusion layers of Pt-CC, Pt-CM and AC-MM were analyzed before and after water pressure of 130 cm, 180 cm and 300 cm (Fig. 3).

Some cracks can be observed on the diffusion layers of Pt-CC and Pt-CM on the surface of air-facing side. After withstanding water pressure, the cracks were torn open and some holes and

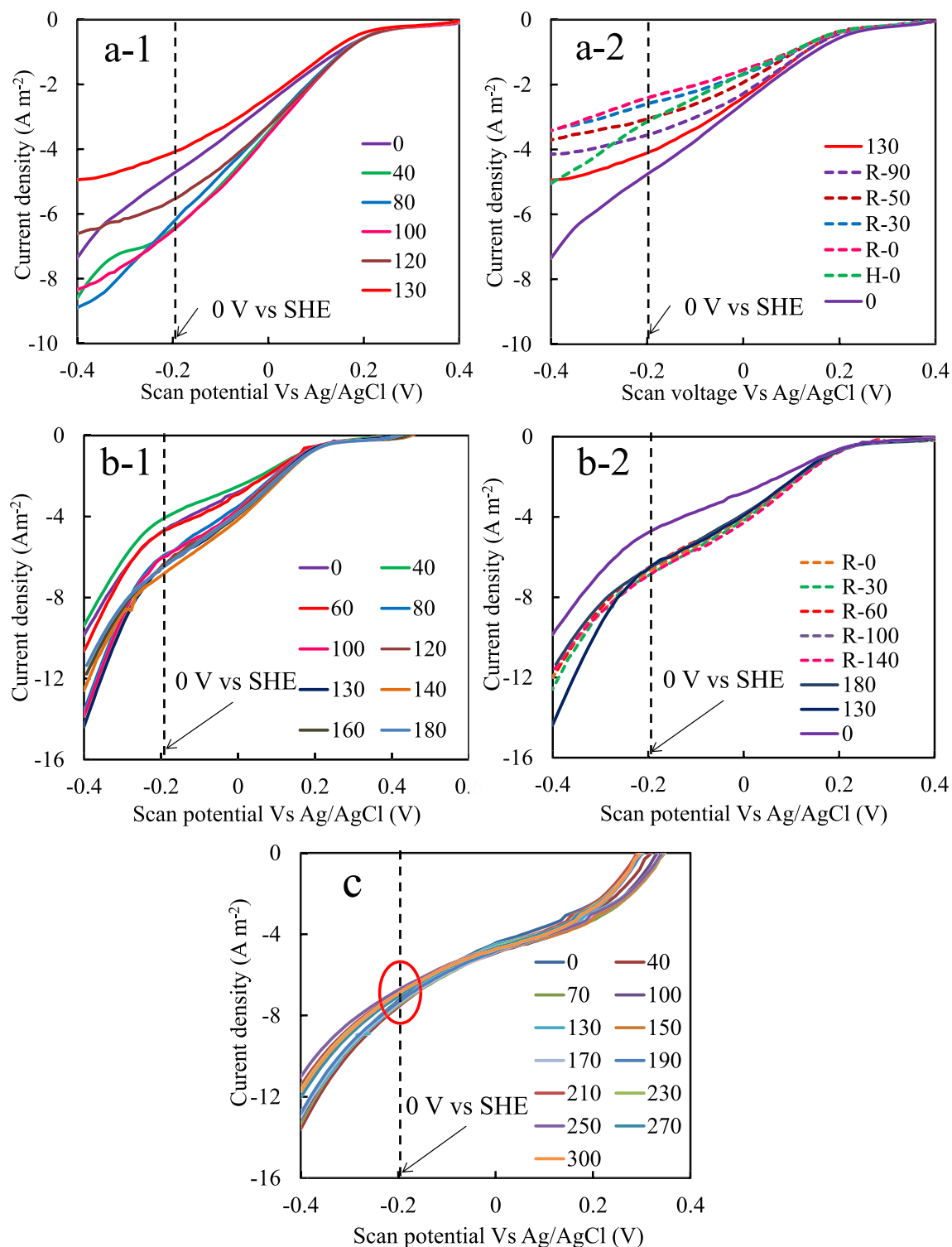


Fig. 2. The effects of water pressure on different kinds of cathodes. (a-1: The performance of Pt-CC under different water pressure; a-2: The loopback tests of Pt-CC; b-1: The performance of Pt-CM under different water pressure; b-2: The loopback tests of Pt-CM; c: The performance of AC-MM under different water pressure. The solid line: the LSV curve in background tests; The dashed line: the LSV curve in loopback tests.)

bulges appeared on Pt-CC and Pt-CM (Fig. 3 A, B). The micrographs in small scale indicated that the stretch of cathodes caused the deformation of diffusion layers in 5 μm scale. The forming of micron size cracks may enhance the oxygen diffusion of the cathodes and consequently result in the increase of current densities (Fig. 4 A, B). However, the serious lacerations of diffusion

layers under higher water pressure may cause the water flooding and prevent the mass transfer of oxygen on the air side of cathodes.

The 300 cm water pressure did not cause obvious changes on the surface morphology of AC-MM. The electron micrographs of AC-MM exhibited that the rolling diffusion layers were more

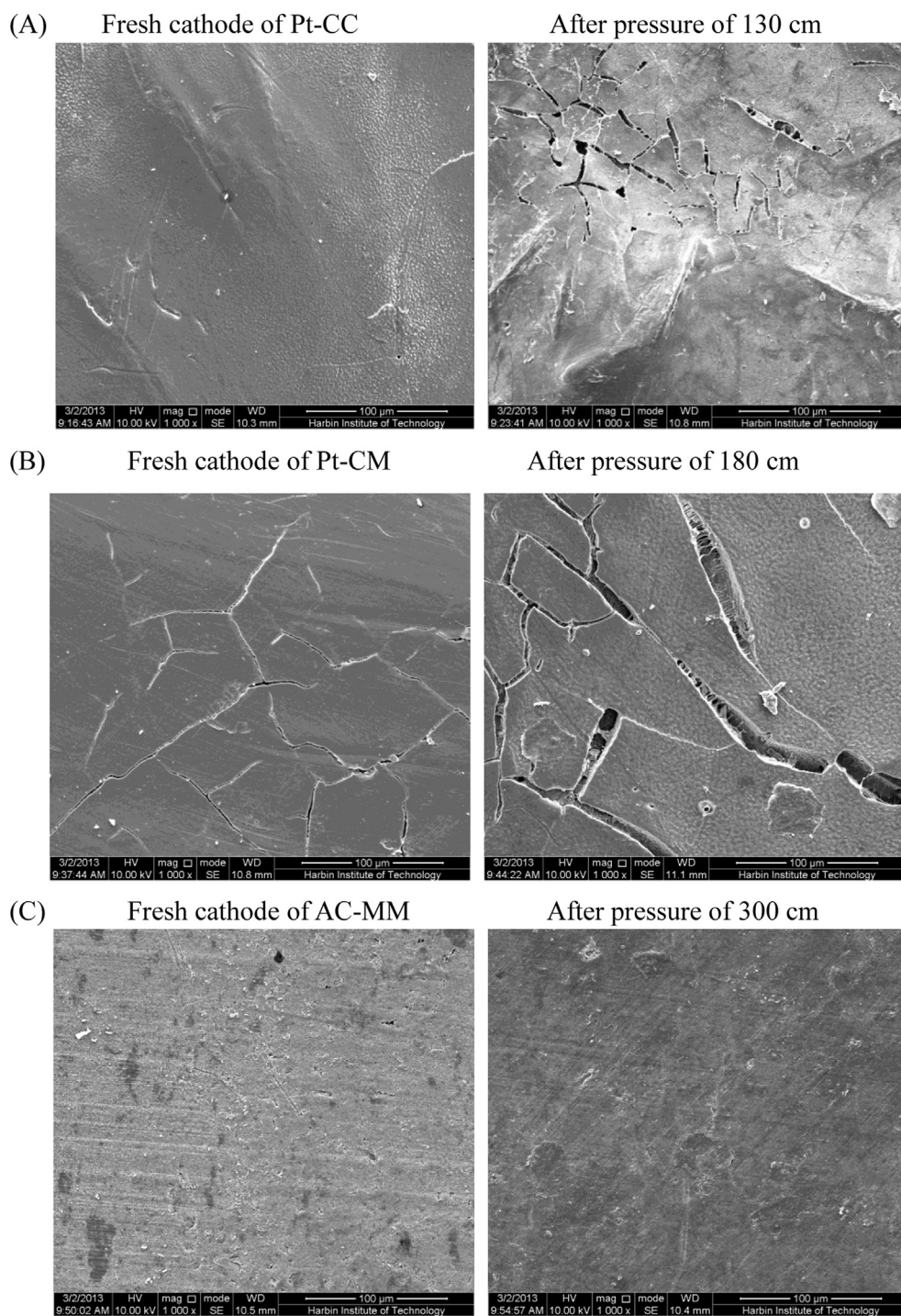


Fig. 3. Scanning electron microscopy (SEM) images (100 μm scale) of (A) Carbon cloth cathode, Pt-CC (B) Carbon mesh cathode, Pt-MM and (C) Rolling stainless steel mesh activated carbon cathode, AC-MM.

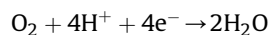
uniform and smooth than that of Pt-CC and Pt-CM and remained unchanged after the water pressure tests (Fig. 3 C, Fig. 4 C).

3.3. The mechanism analysis of cathode performance under water pressures

It is obviously that there existed a water pressure turning point for Pt-CC or Pt-CM. A successive and significant increase of current densities from LSV can be observed before the turning point and

then kept almost stable till the diffusion layers were damaged. The impacts were almost retained after the elimination of the water pressure.

In an air cathode MES, the cathodic reaction could be written as:



The cathodic reaction rate could be affected by changing the concentration of reactants or the number of catalytic sites in the catalyst layer. In this test system, 50 mM phosphate buffered

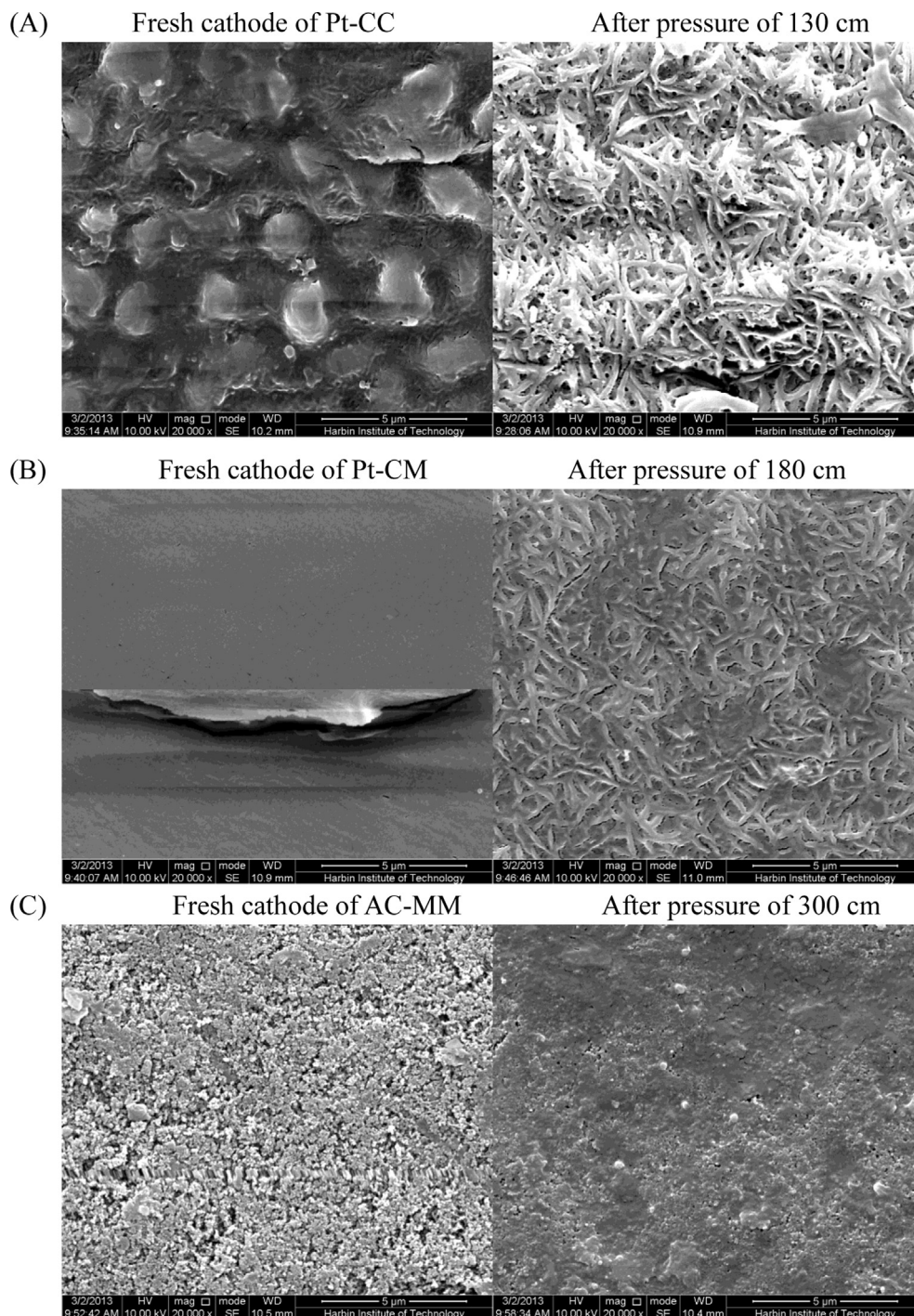


Fig. 4. Scanning electron microscopy (SEM) images (5 μm scale) of (A) Carbon cloth cathode, Pt-CC (B) Carbon mesh cathode, Pt-MM and (C) Rolling stainless steel mesh activated carbon cathode, AC-MM.

solution was used as electrolyte to remain the proton content constant. It can be estimated that the content of dissolved oxygen should influence the cathode reaction. Normally oxygen partial pressure in the atmosphere and the diffusion coefficient of the cathode are two important factors affecting the cathodic reaction. If the structure of cathodes can keep stable under water pressure, as the AC-MM, the cathode performance is unlikely to be affected. So, the hydrophobic diffusion layer was considered to be the key point that affects the water pressure tolerance of cathodes. However, from the laceration of diffusion layers (SEM images) on suppressed

Pt-CC and Pt-CM under water pressures, a mechanism about the process of lacerations and how this process was associated with the performance of the cathodes were demonstrated in this study.

There existed the lateral force (F) generated by water pressure on cathode surface, which can be calculated from the equation, $F = P \times S$, where P is the water pressure and S is the cathode surface area. Under this lateral force (F), cathodes had to stretch and generate a stretch force to balance the lateral force. Since the cathodes were bolt to the reactor on the edge, cathodes had to bend to overcome the lateral force (F). In this system, the lateral

force (F) was determined by the water head. But different cathodes deform in varying amplitude for their diverse mechanical properties under certain water head. The diffusion layers of cathodes have to change their shape during the extension of cathode. In small amplitude of deformation, the stretch of diffusion layers might form more micro-size cracks and holes, which could enhance oxygen transfer. In large amplitude of deformation, the diffusion layers were lacerated in the weak or defective points, resulting in the leakage on the air side of the cathodes (Fig. 5). This model explained why there existed the maximum endurable water head for Pt-CC and Pt-CM.

The curvature degree of different cathodes under the same water pressure relates to their different mechanical properties (Table 1). The mechanical property of cathodes was considered to be another key point that affects the water pressure tolerance of cathodes. As part of cathodes, the catalyst layers and diffusion layers also have some contribution on the mechanical properties, even though the cathode base materials played the decisive role among each part. The whole cathodes including every part were tested for the mechanical properties. Elastic modulus of AC-MM was $4 \pm 0.4 \times 10^3$ MPa, which was over 10 times larger than that of Pt-CM and over 60 times than that of Pt-CC. Larger Elastic modulus meant the great tendency to deform elastically in smaller amplitude and form less broken cracks on diffusion layers under certain tensile stress based on the proposed model. Yield Stress is another important factor, which describes the stress at which plastic (permanent) deformation begins and elastic deformation stops. When the cathodes bend and stretch to overcome the water pressure, if the stress of the cathodes becomes larger than the yield stress of the tested cathode, the irreversible deformations will occur. So the yield stress directly influences the maximal endurable water head of the cathodes, because the permanent deformation could result in the irreversible electrochemical performance changes of cathodes. The ultimate strength is the maximum stress the cathode material can withstand. If the stress of the cathodes under certain water head approaches the ultimate strength, the lacerations and water flooding of the cathode will start to occur in the weak points of cathodes. And if the stress of the cathodes keeps on increasing, the cathodes will be ruptured and damaged, resulting in serious leakage (Fig. 5).

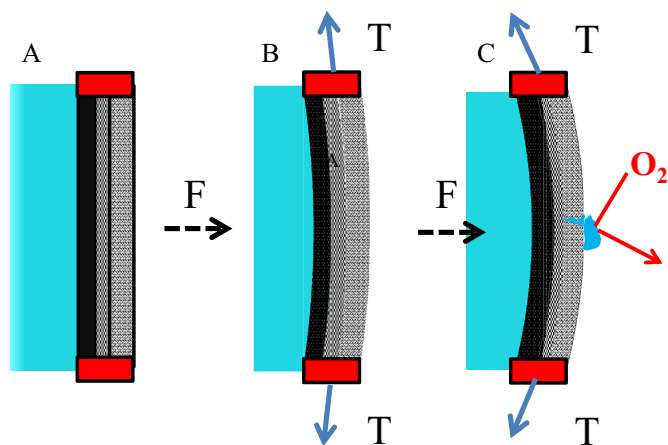


Fig. 5. The illustration of the stretch bending cathode under rising water head and the water flooding of diffusion layer under high water pressure. F : the water pressure force; T : the tensile stress generated in cathode to overcome the water pressure force. A: The shape of cathode keeps stable under 0 cm water head. B: The cathode under low water head. The cathode has to bend and stretch to balance the water pressure. C: The substantial deformation of cathodes under high water pressures caused the lacerations and water flooding.

Table 1

The tensile test of three cathodes (Unit: MPa).

	Elastic modulus in direct stress	Ultimate strength	Yield stress
Pt-CC	64 ± 0.7	8 ± 2.0	1.6 ± 0.5
Pt-CM	$4 \pm 0.5 \times 10^2$	27 ± 3.5	6 ± 3.0
AC-MM	$4 \pm 0.4 \times 10^3$	39 ± 2.6	23 ± 1.3

In this study, AC-MM showed the best water pressure resistance properties. The AC-MM had the highest yield stress (23 ± 1.3 MPa) and ultimate strength (39 ± 2.6 MPa), which meant AC-MM was more flexible than Pt-CC and Pt-CM (Table 1). The flexible and uniform diffusion layer prevented AC-MM from lacerations in stretch bending, even though the formation of micro-size cracks were not detected as well (Fig. 4 C). Meanwhile, the capillary pressure of the porous activated carbon layer has some contribution to the water pressure tolerance of AC-MM as well.

From this mechanical model, the reasons why different cathodes may have different electrochemical responses (Fig. 2) under certain water head could be interpreted. The cathodes of Pt-CC had the lowest yield stress point (1.6 ± 0.5 MPa), which indicated the Pt-CC cathodes were more likely to be affected by water pressure. Under the range of 0–40 cm, the impacts of water pressures were observed on Pt-CC, while the Pt-CM and AC-MM were relatively stable. Under the whole test range, the permanent (irreversible) deformation was observed in the loopback tests of both Pt-CC and Pt-CM, which indicated that the stress caused by the water pressure may exceed their yield stress point during their respective test range. But the ultimate strength of the Pt-CM (27 ± 3.5 MPa) was higher than that of Pt-CC (8 ± 2.0 MPa), which prevent the Pt-CM from serious lacerations and water flooding of the diffusion layers. The serious lacerations of diffusion layers may consequently result in the degradation of electrochemical performances under higher water pressure (Table 1). In the AC-MM, the stress generated by 300 cm water head even did not reach the yield stress point of AC-MM, since no permanent (irreversible) deformation was caused under the whole test range of water pressure. The high mechanical properties of AC-MM resulted in the stable electrochemical behavior under the water head of 0–300 cm.

So it can be concluded that the water pressure tolerance of cathodes depends on the maximum deformation amplitude of the cathodes before the lacerations of diffusion layers and the stretch force provided by the cathodes under this deformation amplitude.

4. Conclusions

Cathodes operated under water pressures were forced to deform. Finite deformation may contribute to the oxygen diffusion increase of cathodes. However, substantial deformation under water pressures higher than the cathode tolerance irreversibly caused the lacerations and water flooding of diffusion layers. Compared to Pt-CC, higher strength substrates like carbon mesh of Pt-CM can reduce the lacerating risk of diffusion layers. The AC-MM functioned stable under the water pressure of 30 KPa and exhibited the best water pressure tolerance due to the best mechanical properties.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data related to this article can be found at <http://dx.doi.org/10.1016/j.jpowsour.2014.05.028>.

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